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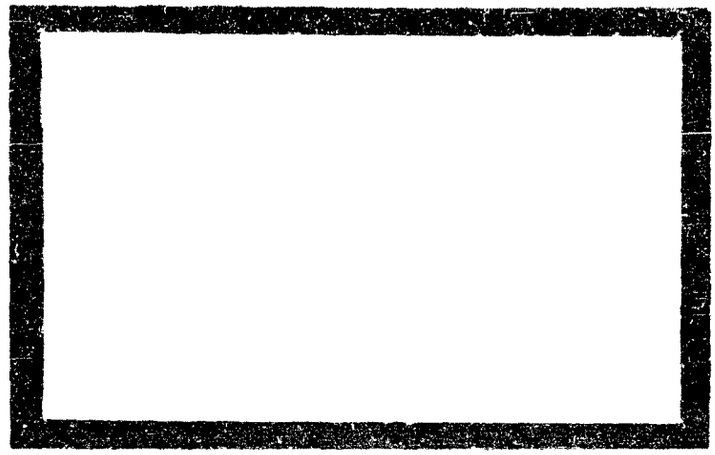

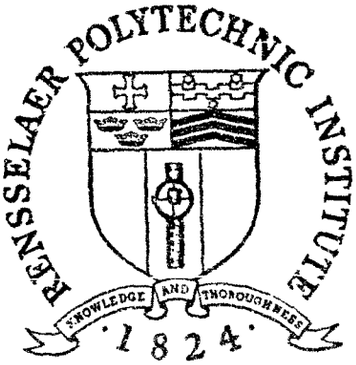

Rensselaer Polytechnic Institute

DEPARTMENT OF METALLURGICAL ENGINEERING

Troy, New York

FINAL TECHNICAL REPORT
Contract Number: DA-30-115-ORD-140
W. A. L. Expenditure Order: 91219-09
O. O. Project Number: TB4-161 D

Title of Project-
QUALIFICATION TEST FOR PLATERS
OF CHROMIUM

FINAL TECHNICAL REPORT

Contractor: Rensselaer Polytechnic Institute

Agency: Office, Chief of Ordnance, ORDTB

Ordnance District: Rochester

Contract Number: DA-30-115-ORD-140

W. A. L. Expenditure Order: 91219-09

O. O. Project Number: TB4-161D

Title of Project: Qualification Test for Platers of Chromium

Object:

To develop a qualification test for Chromium platers and to develop a test for the evaluation of the quality of the chromium plate.

Abstract:

It was found that before a qualification test could be developed, a quantitative method for the evaluation of plate adhesion was necessary. The requirements are that the test should be simple, quick, non-destructive to the ordnance materials being tested, and quantitative. A test for the quantitative evaluation of adhesion, called the scratch test, has been developed, after a search through literature showed that no test suitable to the requirements was available. Because the scratch test does not simulate service conditions that occur in plated ordnance materials, a second test, called

the blast test, was designed and used to show that the scratch test does predict results of the use of plated articles in service. After the development of the adhesion tests, work was initiated in the development of a situation to test the ability of chromium platers. Six initial tests were made and the results are discussed. These results show that a qualification test for chromium platers is feasible.

Conclusions:

1. The scratch test is a quantitative test for the adhesion of chromium plate on steel. Its results, although relative, are capable of predicting the adhesion of chromium plate during severe service.

2. The scratch test is easily and quickly performed, and may be applied in a non-destructive manner.

3. The variables, which are not related to adhesion but do affect the chip depth as read in the scratch test, are plate thickness and angle of scratching tool. Compensation for these effects can be easily accomplished in applying the test.

4. The scratch test should prove valuable in control of mass production plating and in development of new plates and plating processes.

5. Though less easily applied than the scratch test, the blast test will adequately test the adhesion of chromium plate on steel.

6. A qualification test for chromium platers may be designed to show the platers' capabilities.

Recommendation:

It has not been possible within the term of operation under this contract to give the scratch test a thorough production line check. It is recommended that this be done before the test becomes a part of any specification for adhesion.

Report Period:

This report covers the period of July 9, 1951 to September 8, 1952.

Acknowledgments:

Many thanks are due to Dr. P. R. Kosting of the Watertown Arsenal Laboratory and other personnel of the Ordnance Department for helpful suggestions during the progress of the work.

Authors:

Wartan A. Jemian and Arthur A. Burr.

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SCOPE OF REPORT

The material presented in this report represents the work on the development of specifications for the qualification of chromium platers. It is presented under five headings, as follows:

1. Introduction
2. Review of possible adhesion tests
3. The scratch test
4. The blast test
5. The qualification test for chromium platers

INTRODUCTION

The plating of chromium on ordnance materials to improve the performance of the plated part has been developed to a point that the plate will consistently have acceptable qualities if the proper operating conditions are maintained. However some of the plated parts have proved to be poorly adherent in service. The causes of poor adhesion can, in general, be attributed to negligence in operating procedures. Some of the notable causes of poorly adherent plates are:*

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* From private communication with Mr. Robert Hill.

1. Poor grease removal prior to application of the stop-off material, thus allowing the grease to flow onto the plating surface from under the stop-off material when the piece is immersed in the warm bath.
2. Using the same plating solution repeatedly, without cleaning, thus allowing the solution to contaminate the plating surface.
3. Allowing too much time to elapse, for purposes of inspection, between the cleaning and plating operations. During this period the plating surface has no protective coating and an oxide film can form on it.

Now that dependable production line methods have been established, it has been found necessary to be able to test the ability of a contractor or plater to produce a plate to meet the requirements of service. In addition it is desirable to have a means of evaluating the work from the production line.

As the first step in this research on the development of a test or tests to fulfill the needs, it was found necessary to have a means for definitely evaluating plate characteristics and quality. In previous specifications, the emphasis is laid on the plate thickness, distribution, and appearance. It will also be of value to include a quantitative test for adhesion. Such a test has been developed in this

laboratory and specifications for its application are given in Appendix A. The development of this adhesion test, called the "Scratch Test", is described in a later section of this report. The design of a suitable plating situation, for the testing of chromium platers, has been placed as the second and final step in this research and is also described in this report.

REVIEW OF POSSIBLE ADHESION TESTS

A number of adhesion tests have been developed for testing electroplated metal, but they are not adaptable to testing hard chromium on steel.[5,6] The requirements of a suitable adhesion test are that it should be quickly performed, easily evaluated, and non-destructive to the material. The Ollard[1] test, for example, which has yielded the most quantitative results of all the adhesion tests, is impractical because it is necessary to plate to a thickness of at least one-tenth of an inch, and then the specimen must be machined to a very close tolerance for the final test. Even if it were possible to prepare a specimen of the proper thickness and to machine it, this test would fail to be useful because the material being tested would be completely destroyed.

Another type of test for adhesion has been designed especially for chromium plate on steel, but this test is not quantitative. This is the bend test. It is used in many forms in various plating shops, and an example of specifications

for its application follows.

"Test panels, approximately 3 x 6 x 0.04 inch of the same metal as the articles being plated shall be used. Chromium shall be deposited to a thickness of not less than ... 0.002 inch... The test panels shall simulate as closely as practicable the composition, surface conditions, and surface contamination of the part... To determine adherence of the plating the test specimens shall be bent through an angle of 180 degrees on a diameter equal to the thickness of the specimen, straightened, and then carefully examined at 4 diameters magnification for evidence of non adherence." [2]

A bend test similar to the above was first tried in this laboratory, but it was found to be too difficult to interpret the results quantitatively. Another drawback to this type of test is that it can not easily be applied to all types of materials, and will completely destroy those articles to which it is applied. Another type of test tried was the break test, however this test, too, fails in the same respects as the bend test. In this test, the chromium was plated on one surface of shim stock, and then the shim stock was broken with one sharp bend. The principle of this test is similar to that of the bend test in that the amount of plate separated from the shim stock would be a measure of the adhesion, or lack of adhesion, of the plate. For the previously mentioned reasons and especially for the reason that it was desired to have a straight edge, upon which the

measurements of plate separation could be based, this test was eliminated from further consideration.

The test that fulfilled this last requirement, and those of ease in preparation and evaluation, is the scratch test. It is considered that the scratch test is non-destructive, but further tests must be made before this can be established. The scratch test consists of scratching through the plate, well into the base metal, and observing the degree to which the plate has been separated from the base metal along the edge of the scratch. From the first, this test showed promise of good results, and a standard procedure for conducting it has been developed in this laboratory. This procedure will be discussed in detail in a later section of this report.

None of the previously discussed tests simulate actual service, but a test has been designed for this purpose. It is called the blast test. The development of this test is described in a later section of this report.

THE SCRATCH TEST

The scratch test is intended purely as a means for measuring the adhesion of chromium plate to the base metal. It is not intended that this test should simulate actual service conditions. However it has been shown, as explained later, that the scratch test results will predict service results.

The theory of the scratch test is that some of the plate will be separated from the edge of the scratch if the bond between the plate and the base metal is weak. No attempt has been made to determine the mechanism of the plate failure by means of this test. The importance of this test is that it will provide a means of readily determining whether or not a plate is sufficiently adherent to meet service requirements. The results are purely comparative.

This test does meet the requirements for an acceptable adhesion test, with some doubt about the requirement concerning destructivity. This characteristic of the test depends on its application. Generally the scratch test must be considered to be destructive to the materials being tested, however for certain specific cases it may be used non-destructively. It may be possible to attach specimens to the article, which is to be plated, or it may even be possible to find portions, of the article itself, which will not be harmed by the small scratch that is required.

To perform the test, a scratch, which penetrates into the base metal, is made on the plated surface. A perfectly adherent plate should shear off evenly with no chipping, and actually some portions of scratches on well adherent specimens do appear in this condition, at a magnification of 100X, but there have always been at least a few chips at random locations. With weaker bonds, between plate and base metal, not only will more chips appear along

the edge, but they will also extend further from the edge of the scratch.

The chip depth, which is taken to represent the characteristic adhesion value of the plate to base metal, is the distance between the edge of the scratch, at the boundary of the plate and base metal, and the point, of the exposed base metal, which is the furthest from this boundary. A chip is the exposed portion of the base metal from which a part of the plate has been separated due to the scratching operation. This measurement, instead of that of chip area or length along the edge of the scratch, was chosen because of the ease of measurement. If any other type of measurement were made, it would be necessary to photograph at high magnification before measuring. This would require too much time, trouble, and expensive equipment that might not be available in all plating shops.

Figure 4 shows a portion of a scratched specimen, cut off perpendicular to the scratch, through the deepest portion of the second deepest chip along the scratch. The deepest chip is shown adjacent to the second deepest, although this is not usually the case. The dashed lines AA' and BB' are drawn parallel to the scratch at the peaks of the second deepest and deepest chips, respectively. The measurement that is made of the chip depth is also shown. Figure 4 is not drawn to scale, but all of the important angles and dimensions are marked.

Various techniques have been used to derive adhesion values of chromium plate on steel, using the scratch test. The most suitable procedure, from considerations of reliability of results and speed in operation, is outlined below.

To find the characteristic value for a specific set of conditions, five to ten separate specimens should be treated simultaneously in the following manner:

1. Polish the specimens.
2. Clean and plate the specimens under the desired conditions.
3. Scratch the specimens, using a tungsten carbide tipped scratching tool.
4. Measure the chip depth to derive the characteristic adhesion value of the plate, using a metallurgical microscope, which has been equipped with a filar micrometer eyepiece.

A detailed description of the equipment and procedures used in this investigation is given in the following paragraphs of this section. Complete specifications for the scratch test and its application are given in Appendix A.

The plating fixtures used for preparing the specimens were made of copper and lucite. Each rack consists of a 1/4 x 1/2 x 15 inch copper bar, one end of which is bent into a hook for hanging on a bus bar. A hole is bored in the other end for bolting on the specimen, and near the hook, a piece of lead is connected by an insulating piece of lucite.

Figure 1 is a drawing of the plating rack with both the specimen and anode attached. The anode electrical connections are made by a piece of cable from the upper end of the anode to another bus bar.

The lead anode is approximately 3/4 inch away from the specimen. During plating, all parts of the rack and anode, that may be below the level of the plating solution, are masked, except for the surface of the specimen and the corresponding portion of the anode. These racks have been found to be very easy to use and dependable.

Several different types of scratching tools have been tried. One of the first was a diamond tipped phonograph needle, which was found to break too easily. The phonograph needle was tried because its dimensions are standard and well controlled. It was desired to have some tool which could easily be reproduced in any other laboratory, by other workers who may wish to use this test.

Because the diamond was found to be too brittle, a tougher material was sought. Tungsten carbide was tried because of its toughness and availability in many different and useful forms. Two forms were chosen. One was a carbide edged cabinet scraper blade, and the other was a carbide tipped lathe tool.

Each of these tools has its special applications to this test. The carbide edged cabinet scraper is adaptable to scratching inside curved surfaces of small radius because

it is so thin. This tool was used in making the scratches on the specimen of electroplates on cylindrical surfaces. The cabinet scraper blade consists of a steel plate, approximately 1/8 x 1 x 3 inches, with a strip of tungsten carbide cemented along one of the long edges. The dimensions of the carbide strip are approximately 1/8 x 1/8 x 3 inches. The carbide tipped lathe tool is more adaptable to scratching surfaces where no restriction has to be placed on the size of the tool. It can be easily mounted in a machine for mechanical scratching and held in a lathe tool holder for hand scratching. A lathe tool holder makes a convenient handle. Actually any carbide tipped tool will serve the purpose, provided that it may be properly shaped and held.

The scratching tool was designed to make a "V" shaped groove of 120° included angle between its sides. This angle, which is designated as angle alpha in Figure 2, was originally chosen in order to extend the life of the tool, and later measurements with scratching tools have shown that there are other considerations that favor the 120° included angle. In Figure 2, which shows the geometry of a scratching tool, direction D is the direction that the tool is moved to make the scratch and angle rho is the rake angle.

The radius of curvature of the point of the tool is not critical, with the exception that it must be sufficiently small so that the tool can easily penetrate far enough into the base metal when the scratch is made. The portion of the

tool that shears the interface of the plate and base metal must be straight in order for the results of this test to be reproducible.

The microscope used for the measurements is a metallurgical microscope, whose standard eyepiece has been replaced with a filar micrometer eyepiece. Figure 9 is a photograph of the microscope used for these measurements. This eyepiece has a travelling hairline, whose position is controlled by a graduated knob. All measurements made have been recorded in filar units, which is a linear dimension and which may be converted to centimeters or inches if desired. For example, one filar unit equals 0.0038 inches with the apparatus used in this laboratory. However, this conversion has not been used because the results of the scratch test are comparative.

In this laboratory, the specimens have been prepared by grinding them on a belt grinder to a belt grit of 400 and then in some cases to metallographically polish and etch, alternately, several times. This last operation both provides a smoother surface and removes the layer of distorted metal which was left by the grinding operation. For one particular set of measurements the plates were ground parallel on a surface grinder.

It was found to be very difficult to consistently obtain poor adhesion when the surfaces of the base metal had been polished and etched. Therefore in order to obtain a

greater variety of adhesion values in the specimens used for the correlation of the blast and scratch tests, the layer of disturbed metal was not removed. In any case, a thorough reverse etch treatment will remove this layer.

Another method used for varying the adhesion of the plate was to remove the protective coating of oil to various degrees. This was accomplished by omitting or varying one or more of the cleaning procedures so that slight contamination might remain on the surface when the specimen is plated. A thorough discussion of surface contamination is given in the reports of an AES sponsored research[3] conducted at Columbia University. These papers report a method of testing for and controlling various degrees and types of surface contamination. In general, their method, too, is to first thoroughly contaminate the surface and then clean to different degrees.

It is an interesting fact that all of the specimens had a similar appearance after being plated, regardless of their adhesion properties. Of course there was a marked difference in appearance between plates on ground and on polished surfaces. This is due, entirely, to the influence of surface smoothness of the base metal.

In making the scratch, the tool should scratch the specimen once, in a straight line of uniform depth. If a scratch is found to be too shallow, it should not be re-scratched. Instead, a new scratch should be made on another portion of the specimen.

The thinner plates may be scratched by hand, using some guide to insure a straight scratch, although the guide is not absolutely necessary. In this laboratory the specimens have been clamped rigidly, with a short piece of angle iron, in a vise. The angle iron, which has been surface ground for smoothness, guides the tool. With this arrangement, no difficulty was encountered in making a straight and uniform scratch.

In the studies of the effect of plate thickness on the scratch test, it was found necessary to make the scratches mechanically, due to the greater thickness of some specimens making it too difficult to scratch by hand. For this operation, a special apparatus was designed and built. The principle of its operation is that the specimen is moved over the scratching tool, which is held between the ways that guide the specimen. Figure 8 is a drawing of this guide for mechanical scratching. The small groove, between the ways is provided so that any roughness, caused by the scratching operation, can not interfere with the motion of the specimen and so that the chip depths will not be influenced by friction on the ways.

The scratching tool is mounted between the ways as shown in the drawing. It is backed, in the hole, by a slotted bolt and after the tool is adjusted into the correct position the tool clamping bolt is tightened from the front. The height of the tool point, above the ways, is controlled

by the tool adjust bolt. The square end of the tool rests on the end of the bolt, which is tapered. Therefore, as the bolt is moved in or out, the point of the tool moves up or down, respectively.

The force to move the specimen over the scratching tool was provided by the travelling tool post of a lathe. It had been found to be very difficult and tedious to mount the specimens on any of the standard machine tools so that the scratching tool could make a scratch of uniform depth. Therefore this apparatus, which utilizes the power of one of the readily available machine tools, was designed.

The first measurements made consisted of averaging the depths of fifteen chips taken consecutively along a random length of the scratch. The only restriction imposed on the section that was chosen for measurement was that the scratch must be straight and uniform in depth in this portion. This specification is still held. There are generally twenty chips in the field of view of the microscope, so that the specimen did not have to be repositioned during any measurement. Results from two groups of specimens measured in this manner, are shown in Figure 5.

Figures 5, 6, and 7 are all similar. Their interpretation is that the vertical axis represents the number of specimens in a group, and each horizontal axis is graduated in filar units. Each group of samples, prepared under a given set of conditions is represented by a triangle whose

base lies on the horizontal axis. The peak of the triangle lies above the average value for the group and the two other corners represent the statistical variance of the group. This is a measure of the range of the values. The height of each triangle represents the number of specimens in each group. The data that these figures are based on are listed in Tables 3, 4, and 5.

Figure 6 represents two groups measured by a greatly condensed method in which the three largest chip depths are averaged. It is evident that the results shown here are not comparable to those shown in Figure 5. The reason is that different base metals were used in the two instances. Figures 6 and 7, however, are based on results taken from specimens of the same base metal. Hence the mean for the well adherent plate groups are comparable. Some variation is to be expected though, because the two groups in Figure 7 were derived by measuring the depth of the second largest chip that could be found, in contrast to those of Figure 6 which were derived from the three deepest chips. In the case of each figure, the group with the smaller mean adhesion value is the group that has been well cleaned prior to plating.

As the method has become abbreviated, the variance in the results has increased, but the separation between the group averages has also increased. Measurements have been made on several scratches per single plate, but they are not as reliable. If larger surfaces were tested, such methods

would be of value.

Because all of the measurements have been made on small specimens this abbreviated procedure has been used throughout the work in this laboratory.

Measurements have been made to determine the influence of two variables on the results of the scratch test. These variables are plate thickness and angle of the scratching tool. In the preliminary investigations, values for these variables, had been chosen without definite knowledge of their effect.

It has been assumed that the thickness of the plate on the specimen would have no bearing on the bonding of the plate to base metal, although the results of the scratch test would be affected by the plate thickness. The plate thickness, 0.0005 inches, that is marked in Figure 4 is what has generally been used. Measurements have been made on plates of various thicknesses and the results are reported graphically in Figure 10. The base metal for these specimens was tempered martensitic steel.

These specimens were surface ground parallel for uniformity. They were all given proper cleaning treatments prior to being plated in order that each plate would be well adherent. The thicknesses were varied by varying the plating time. The data for these specimens are given in Table 6.

The curve in Figure 10 is a graph of chip depth, D, versus plate thickness, T. The line, $D = 0.00581\ T + 0.289$,

was fitted so that it is as close as possible to each of the points in the array. Namely, the sum of the squares, of the vertical distances from each point to the line, is a minimum. The method for making the curve meet this requirement is explained in almost any text on statistical methods of analysis and control.[4]

As can be seen from the curve in Figure 10, the chip depth does increase as the plate thickness increases. This result is probably due to the increasing total strength of the plates as their thickness increases.

The specimens used in testing the effect of tool angle were all well adherent. The surfaces were alternately polished and etched, and the recommended cleaning procedures were carefully observed prior to plating. The plates were made simultaneously.

Figure 3 shows the plot of chip depth versus included angle of the scratching tool. The curve was drawn through the means of each set of chip depths, at each angle. Each plate was scratched once with each of the scratching tools, and as can be seen from the tabulations in Table 2, the results on each individual specimen parallel the trend of the mean very well.

The curve indicates that the best point to use will be that having an included angle of 60°, because with this angle the results will be more pronounced. However, the 120° included angle has the advantage that the results will

not be so greatly influenced by a change of angle due to faulty grinding. A small change of angle, in the region of 60° might have an extremely bad effect on the results of measurements. The results would tend to show a better adhesion than is actually the case, perhaps allowing faulty articles to pass inspection. Also, as mentioned before, the tool will have a longer life with an included angle of 120°.

The scratch test has been used principally as a means for comparing the adhesion of plates from various plating baths and of plates made on specimens which have had different surface preparations. The next few investigations are examples of the application of the scratch test.

The first application is in using the scratch test to determine the relative qualities of plate, with respect to the adhesion, made in baths of different compositions. Figure 11 is a graphical presentation of the results of two groups of well prepared specimens. Its interpretation is similar to that of Figures 5, 6, and 7. One group, in Figure 11, was plated in a standard chromium bath (dashed line) and the other in a "Unichrome" bath (solid line). The Unichrome bath is a commercial bath whose sulfate concentration is controlled automatically by salts added to the chromic acid salts. This bath is used extensively by some plating shops for plating small mechanical parts.

One of the features of the bath, other than that the sulfate concentration is automatically controlled, is that the

resulting chromium plate is brighter than that deposited in a standard bath. Although the average chip depth of the Unichrome plate (solid line in Figure 11) is slightly greater than that of the specimens plated in the standard bath, the results are sufficiently close to establish that the Unichrome plate is well adherent.

An example of a second application is in testing adhesion for the development of other types of electroplates. A specimen was submitted by another laboratory. This specimen had essentially the same type of base metal, a tempered martensitic steel, as has been used in this laboratory, but the electroplate was a chromium alloy, instead of pure chromium.

The scratch test results show that the adhesion of the alloy was very poor. The average value was 0.917. The average value for a plate of similar thickness (0.003") should be below 0.600. This result was substantiated by service results.

The third application of the scratch test was in using it as a control test for production plating. Test specimens were plated simultaneously with three components in the production baths operated by a private contractor. Along with each of the three components tested, five specimens were plated. The plate thicknesses were 0.0005 inches, as has been used in this laboratory for most of the measurements. For each component, the specimens were plated consecutively so

that there was a specimen in the bath during almost all the time that the component was in the bath. These specimens had also been given the identical cleaning treatment as that given the components. The results of the scratch test measurements are listed in the table below.

SCRATCH TEST RESULTS ON THREE COMPONENTS

COMPONENT	AVERAGE CHIP DEPTH
A	0.253
B	0.412
C	0.534

The results of these tests show that the adhesion of the three components are not the same. There is no doubt about the adhesion of component A but components B and C will probably not last as long in service as desired. Correlation with actual service has not been possible within the time limit of this investigation.

There are several errors that may affect the results of the scratch test. However, with care, these errors can easily be eliminated. The following is a brief list of the possible errors. They will be discussed as follows:

1. Non-representative Specimen
2. Improperly Made Scratch
    a. Insufficient penetration of tool
    b. Scratch not straight
    c. Non-uniform depth of scratch

3. Misinterpreted Results

a. Broken base metal mistaken for chip

b. Broken chromium plate mistaken for chip

c. Stain mistaken for chip

d. Non-representative chip

1. The first error will most likely arise when the specimen is not an integral part of the material being tested. To eliminate this error, with the greatest amount of certainty, a means must be found to test the plated article itself.

2. A little practice in making scratches will be sufficient to familiarize the inspector with the technique. It will be readily seen if the scratch is not straight, or not of uniform or sufficient depth. If the scratch is found to be lacking in any of these respects, it should be corrected by making a second scratch.

If the depth of the scratch is not uniform, the size and distribution of the chips will be influenced. It was observed that the chips are symmetrically located along a scratch that is uneven in depth. However, along an even scratch, it is coincidence if two chips are oriented symmetrically across the scratch. Another cause of uneven depth may be that the surface of the specimen is not sufficiently smooth. This is controlled during the grinding and polishing of the base metal. The plate will reproduce the original surface.

3. The last group of errors, namely the misinterpretation of results, are the ones most likely to cause trouble. Before measurement of the chip depth, care must be taken to scan the length of the scratch. A chip, where the chromium has separated from the base metal, will generally be well outlined. In many cases, the remaining plate casts a small shadow. The surface of a chip is flat and parallel to the surface of the chromium plate, but at a different level.

A place where base metal has been removed may be easily identified by its gouged appearance. It will be deep, with a rough or rounded bottom, if it has any bottom at all. Generally the sides curve in to meet the side of the scratch.

If a piece of chromium plate has broken away from itself, it may appear as a true chip. The distinguishing characteristics of this type of chip are that the edges are not well defined and the color of the metal will be similar to that of the surface of the plate.

The stains, which can be misleading, may best be controlled by carefully washing the specimen when it is removed from the bath.

If there is any doubt as to just what is a chip and what is not, the inspector may etch the specimen. A quick wash with copper sulfate solution is helpful in distinguishing chromium from steel. However care must be taken that the plate is not left too long in the etch. Better etchants are nital and picral, which darken the steel. With this type of

etchant there is no danger of mistaking areas covered with a thin layer of chromium for exposed areas of base metal. The copper sulfate etch is susceptible to this type of error because it can penetrate through the cracks of the chromium plate and will deposit a layer of copper apparently on the plate itself.

When an inspector has had some experience, it will only take from one half to one minute to locate the representative chip and then only another fraction of a minute to measure it. Choosing the chip that is representative is not difficult. But in order that too much time not be spent, the choice requires a certain amount of judgement by the inspector. Errors, which are bound to occur, can be minimized by proper sampling.

In conclusion, it may be emphasized, therefore, that the scratch test is an easily performed, quantitative adhesion test. The value of adhesion is not expressed in terms of the force required to separate a unit area of plate from base metal, but is a simple linear unit which is relative. This test should find its widest application as a control of production plating.

THE BLAST TEST

The blast test is intended to simulate the conditions that occur in the use of certain ordnance materials. The method is to place a plated specimen in an apparatus in which an explosive may be discharged against the plate. Similar

tests are being used at several research laboratories.

The first trials of this type of test used an apparatus that held specimens of a cylindrical shape. The specimens were threaded steel bars in which tapered holes had been bored. The inner surfaces were plated. The taper provided a choke to concentrate the effect of the exploding gases. The charge used was a blank twelve guage shotgun shell. It was found that a well adherent plate would withstand at least twelve blasts, whereas a poorly adherent plate would be appreciably flaked away within one or two blasts.

It was desired to find a way to correlate the scratch test with this simulated service test. This necessitated the use of flat specimens that would be adaptable to testing by both methods. Therefore tests were made on blast test specimens whose surfaces were normal, instead of parallel to the direction of gas flow. Results similar to those obtained with cylindrical specimens were found with this new positioning of the specimen, so a new apparatus, intended to hold flat specimens, was designed.

Figure 12 shows the new blast test apparatus. The apparatus is made of several blocks of steel that can be bolted together, with four legs, so that it stands and fires upward. The firing mechanism, which is powered by a spring, screws into the bottom block; the next block holds the blank shotgun shell; and the top block, which has a slot, holds

the specimen in position in the slot. The short bolt, which has a hole in its center, screws into the top block to clamp the specimen in place. The specimen has a hole in its center, which is lined up with the hole in the specimen clamping bolt. In order to provide this hole for the gases to escape, the specimens were designed to be mounted, on the plating rack, by a countersunk flat head bolt through their centers.

In order to test a specimen, it is inserted in the slot; centered by a pointed rod that fits the hole in the specimen clamping bolt; then the bolt is tightened and the centering rod is removed. Next, the shotgun shell is put in position through the bottom, and the firing mechanism is screwed in place and cocked. The apparatus is fired by pulling on the string that is attached to the pin in the firing mechanism.

For reasons of safety, the test was conducted out of doors in an isolated spot where the operator was protected by a large brick wall. In practice it was found that the apparatus is safe, but precautions were still maintained.

The shotgun shells were prepared by cutting away the shot holding section and removing the cardboard plugs so that the powder was exposed. This last operation was never done in advance in order that no powder would be lost and so that the powder would remain dry. The shotgun shells were standard 12 guage.

In the derivation of a number to represent the adhesion of the plate, two factors have had to be considered. The first factor is the number of blasts to which the specimen must be subjected and the second is the manner in which the adhesion value is obtained.

Through trial, it has been found that a single blast is sufficient for the test. Figure 13 shows a well adherent plate after it has been subjected to 15 blasts. This plate has remained unharmed except for the tarry deposits left by the blasts. No plate was removed. Figures 14 and 15 show a poorly adherent plate and a very poorly adherent plate, respectively, after being subjected to only one blast each. The damage to the plate is quite severe in both cases. The specimen shown in Figure 14 had not been given a proper cleaning prior to plating and the other had been plated without even removal of the film of oil.

It is interesting to note that great damage, to the plate, occurs on the portion that is not directly exposed to the blast. This occurs because part of the expanding gases escape to the sides of the specimen instead of through the hole in the center. Flames from the explosion come from both the top and the sides of the apparatus.

The adhesion value that is used in this research is called the adhesion ratio, which is the ratio of the area of unremoved plate to the area of the plate originally on the specimen before it was subjected to the blast. The original

plate area is easily calculated and the area of the unremoved plate was found indirectly, for reasons of convenience, by measuring the area of the removed plate with a planimeter.

In order to correlate the scratch and blast tests, groups of ten specimens were prepared under identical conditions so that the adhesion would be uniform within each group. After plating, each group was randomly subdivided into two five-specimen groups, one of which was used for the blast test and the other for the scratch test.

Figure 16 is a plot of adhesion ratio versus chip depth. Each point represents ten specimens which were plated under identical conditions. Five specimens from each group were tested by the scratch test, and the remaining five were tested by the blast test. This curve shows that there is a strong correlation between the two tests. This means that the scratch test is justified as a measure of the adhesion of chromium plate that is to withstand the conditions that occur in some conditions of severe service.

THE QUALIFICATION TEST FOR CHROMIUM PLATERS

A test has been designed for testing the ability of chromium platers. The prime aim of this test is to determine whether or not a person is able to electroplate chromium on steel to specifications. The present state of the test is that of a laboratory test. A reproduction of the instruction sheets given to the applicants is in appendix B.

The plating problem was for the applicant to plate one specified surface (see Figure 17) of a formed steel plate. The plater was supplied with all necessary equipment for preparing the specimen surface, making electrical connections, and maintaining proper operating conditions. The points on which he was tested are:

1. Choice of proper anode
2. Proper positioning of anode
3. Calculation of correct current
4. Ability to determine polarities
5. Ability to follow directions

Figure 18 is a drawing of the cover of the plating bath, to which all parts of the equipment are attached. "P1" and "P2" are lucite posts, on which the cathode should be clipped. The lead wire may be passed through hole "L". "H1" and "H2" are for the stems of the heating tube. "T1" or "T2" may be used for positioning the thermometer. "F" is for the exhaust tube for removing the fumes. 1, 2, 3, 4, 5, and 6 are alternate positions for mounting the anodes. Figure 19 shows the three different anodes from which the applicants could choose. All three anodes are lead. The proper anode is the cylindrical one and its proper position is in hole number 1 in the bath cover. This anode will deposit a visibly poor plate when positioned in any other manner as will either of the other two anodes in any position. When in the proper hole, the surface of the cylindrical anode will be concentric

with the specimen surface. It is left to the plater to decide that it is relatively unimportant that this anode is not truncated. Specimens have been made with each of the anodes in each position and it has been found that the choice and positioning of anodes is as critical as stated and that the shape of the end of the proper anode does not greatly influence the plate that is deposited.

The calculation of the proper current could be done in several ways. The easiest method is to notice that the average height, regardless of the flanges, of the plating surface, is one inch, exactly (as specified in figure 17). Also the length, which was given indirectly, could easily be found to be "pi" (3.1416) inches. Therefore the area of the plating surface is pi square inches. A graph of surface area versus current was provided. The surface area was plotted from 0 to 5/2 pi square inches, in large units of pi/2 square inches, as shown in Figure 20. However, the curve that was given the platers for use in the test, was subdivided further so that the reading that they needed fell on a line. The abscissa was marked in units of pi in order to give a good hint about the proper method of calculation. Also a table (see Table 1) of plating conditions, with proper current density, was given in order that the current might be calculated by slide rule, if desired.

The wiring was complete except for the attaching of a clip lead to each of the electrodes. It can be seen from

the wiring diagram in Figure 21 that there was no danger of the meter being put in backwards. The polarity of the clip leads could be found from the meter, with the knowledge that the positive lead, from the outlet or source, is attached to the positive terminal of any meter. The polarity was not marked in any other way.

There was no way in which the plater could fail to see how the cathode should be attached, because it was given to him, already in position. However, it did have to be removed for preparation.

The test of the platers ability to follow directions was determined by whether or not he maintained the current and bath temperature properly and whether or not he used care in masking off the proper surfaces.

The steel used for the specimens was zinc covered Hull cell plates. These were cut and bent as shown in Figure 17. They could easily be stripped in dilute HCl by the plater.

No attempt was made to apply the scratch test to determine the adhesion of the plates, although this was considered for later tests, using a firmer specimen base metal.

The qualification test was tried on six men, whose plating experience amounted to one brief course in electroplating. The results from these six tests are listed in the table below.

QUALIFICATION TEST RESULTS

ITEM	NUMBER CORRECT
1	2
2	2
3	4
4	2
5	0

As can be seen, the results of this test are very poor. As far as the individuals are concerned, the results range from almost perfection to complete failure. One plater completed every portion of the test properly, except that he was careless in masking the specimen properly. These poor results may be attributed to the inexperience of the men in the fundamental plating practices. They do not realize just what factors are critical and just how critical that they are. Unfortunately there has not been sufficient time to try this test on more experienced platers. It is believed that an experienced plater could easily complete the task.

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3 Linford, H. B. and E. B. Saubestre, AES Research Project No. 12, "Cleaning and Preparation of Metals for Electroplating," PLATING.

I: Critical Review of the Literature, Dec. 1950, p. 1265, January, 1951, p. 60, February, 1951, p. 158.

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IV: Degreasing Evaluation Tests: Sequential Testing, November, 1951, December, 1951, p. 1263.

4 Hoel, Paul G., Introduction to Mathematical Statistics, pp. 78-81.

5 Schlaupitz and Robertson, PLATING, V 39, July and August (1952).

6 AES Research Project Number 3, "Adhesion of Electrodeposits," The Monthly Review 32 September, October, November, and December (1945), 33 January, February, March, June, and December (1946).

TABLE 1

Plating Conditions

The following operating conditions are recommended and used by the arsenals for electroplating chromium on steel. These conditions are also used in the laboratory at the Rensselaer Polytechnic Institute.

BATH COMPOSITION: 2.5 M Chromic Acid (33 oz/gal)
0.025 M Sulphuric Acid (0.33 oz/gal)

BATH TEMPERATURE: 130° F

CURRENT DENSITY: 2.1 amps/sq. in.

Lead anodes are used, with an anode to cathode separation of approximately 3/4 inch.

TABLE 2

This data is used in the curve of Figure 3, which is the graph of Chip Depth vs. Tool Angle. The specimens were prepared simultaneously in a standard chromium bath. The data on the measurements of 120° included angle are also used in Figure 11 to represent results from a standard chromium bath. See Table 1 for the composition of this bath.

SPECIMEN	INCLUDED ANGLE OF TOOL			
	30°	60°	90°	120°
1	.220	.263	.531	.154
2	.282	.244	.268	.236
3	.172	.311	.687	.143
4	.172	.308	.679	.259
5	.319	.378	.542	.246

The following is the data on specimens plated in the Unichrome Bath. These specimens received the same cleaning treatments as the above specimens.

SPECIMEN	CHIP DEPTH
1	.258
2	.324
3	.240
4	.417
5	.366

TABLE 3

Scratch Test Measurements

Each value is the average of fifteen chip deoths along one scratch, in filar units times 10^3. (See Figure 5)

Clean Plates	Oiled Plates
263	267
242	385
252	325
225	377
218	338
253	341
246	399
181	488
191	278
234	340
Mean 231	364
Variance ±24	±51

TABLE 4

Each value is the average of the three largest chip depths on one scratch, in filar units times 10^3. (See Figure 6)

Clean Plate	Oiled Plate
341	651
286	584
407	505
388	631
358	575
348	724
368	1007
337	592
Mean 354	659
Variance ±36	±56

TABLE 5

Each value is the depth of the second deepest chip along a scratch, in filar units times 10^3. (See Figure 7)

	Clean Plate	Oiled Plate
	247	673
	341	929
	295	500
	339	571
	306	422
Mean	306	619
Variance	±48	±235

TABLE 6

The following data is used in Figure 10 for the graph of Chip Depth vs. Plate Thickness.

SPECIMEN	PLATE THICKNESS X 10^4 INCHES	CHIP DEPTH
1	5	.301
2	6	.285
3	7	.174
4	9	.286
5	9	.337
6	9	.367
7	9	.406
8	11	.331
9	11	.300
10	13	.370
11	13	.338
12	13	.604
13	15	.400
14	21	.420
15	22	.394
16	24	.595
17	35	.383
18	35	.416
19	37	.582
20	39	.528
21	41	.449
22	43	.606
23	60	.763
24	63	.700
25	66	.523

TABLE 7

The following data is used in Figure 16, which is the curve of the correlation of blast and scratch tests. Each item under each of the last two columns represents the average value of five measurements.

GROUP	CHIP DEPTH	ADHESION RATIO
1	.271	.889
2	.350	.905
3	.414	.795
4	.433	.795
5	.434	.810
6	.468	.842
7	.551	.718
8	1.022	.464

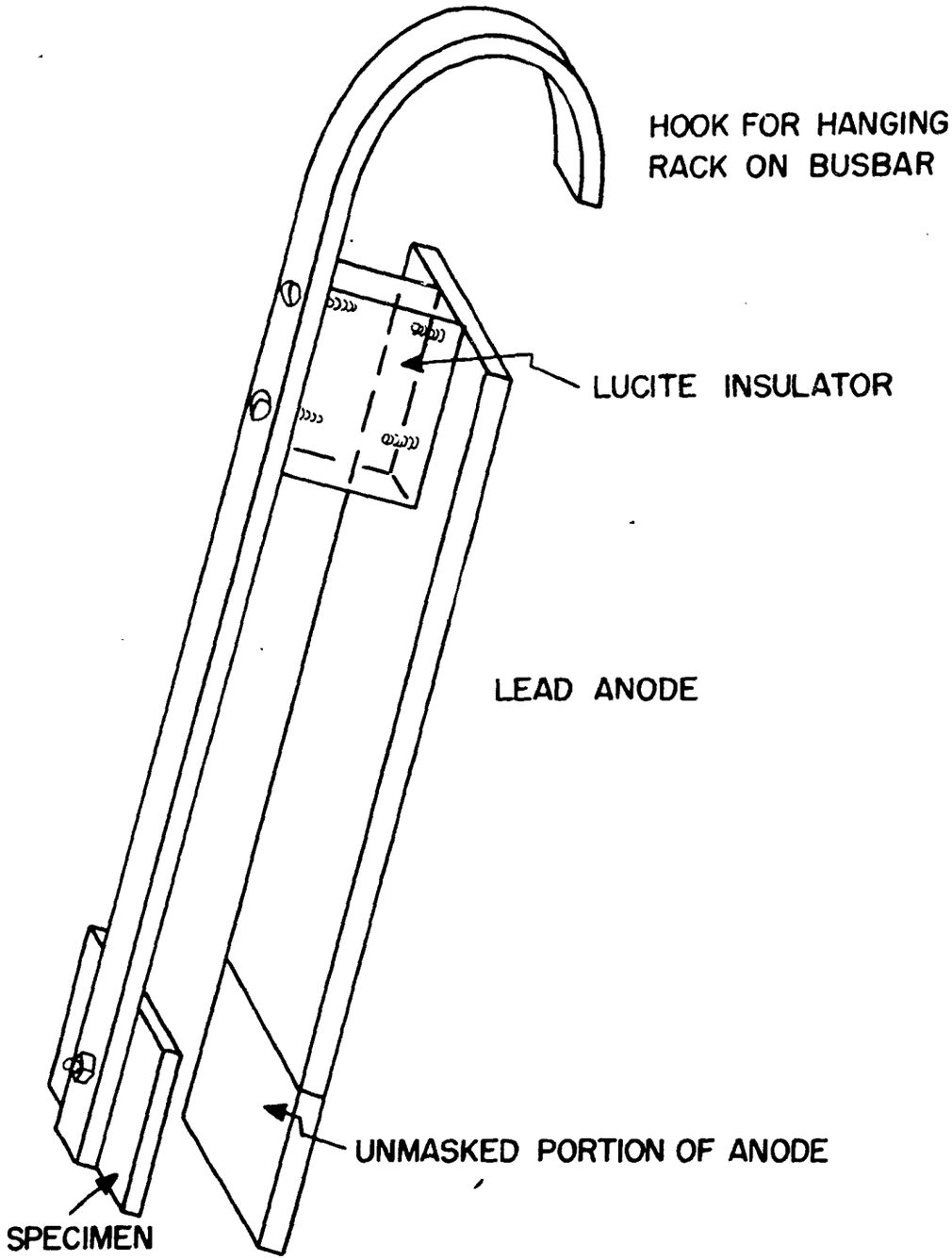


FIGURE 1: PLATING FIXTURE
(APPROXIMATELY 1/2 SIZE)

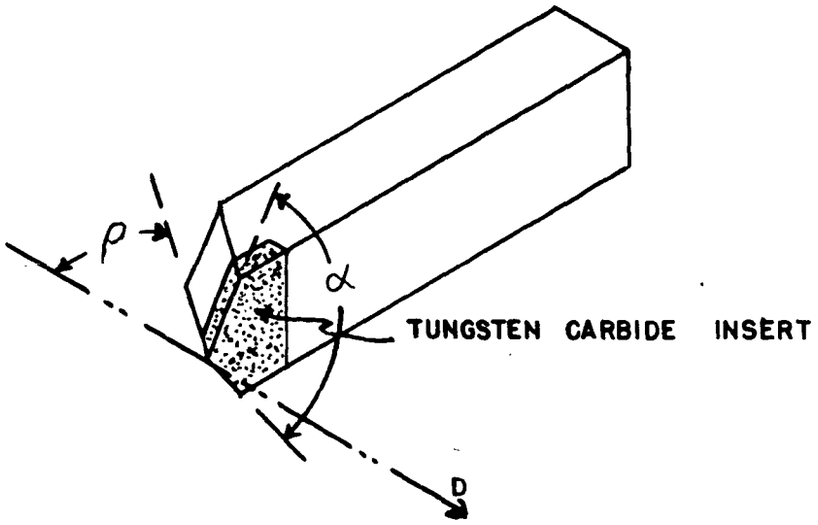


FIGURE 2: THE SCRATCHING TOOL.

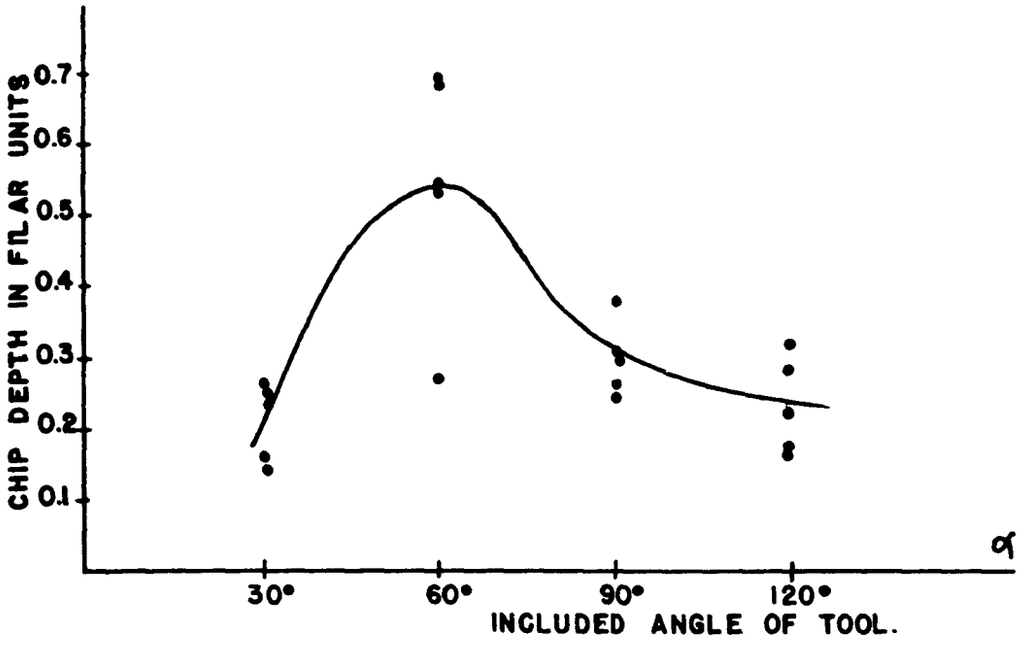


FIGURE 3: EFFECT OF TOOL ANGLE ON CHIP DEPTH.

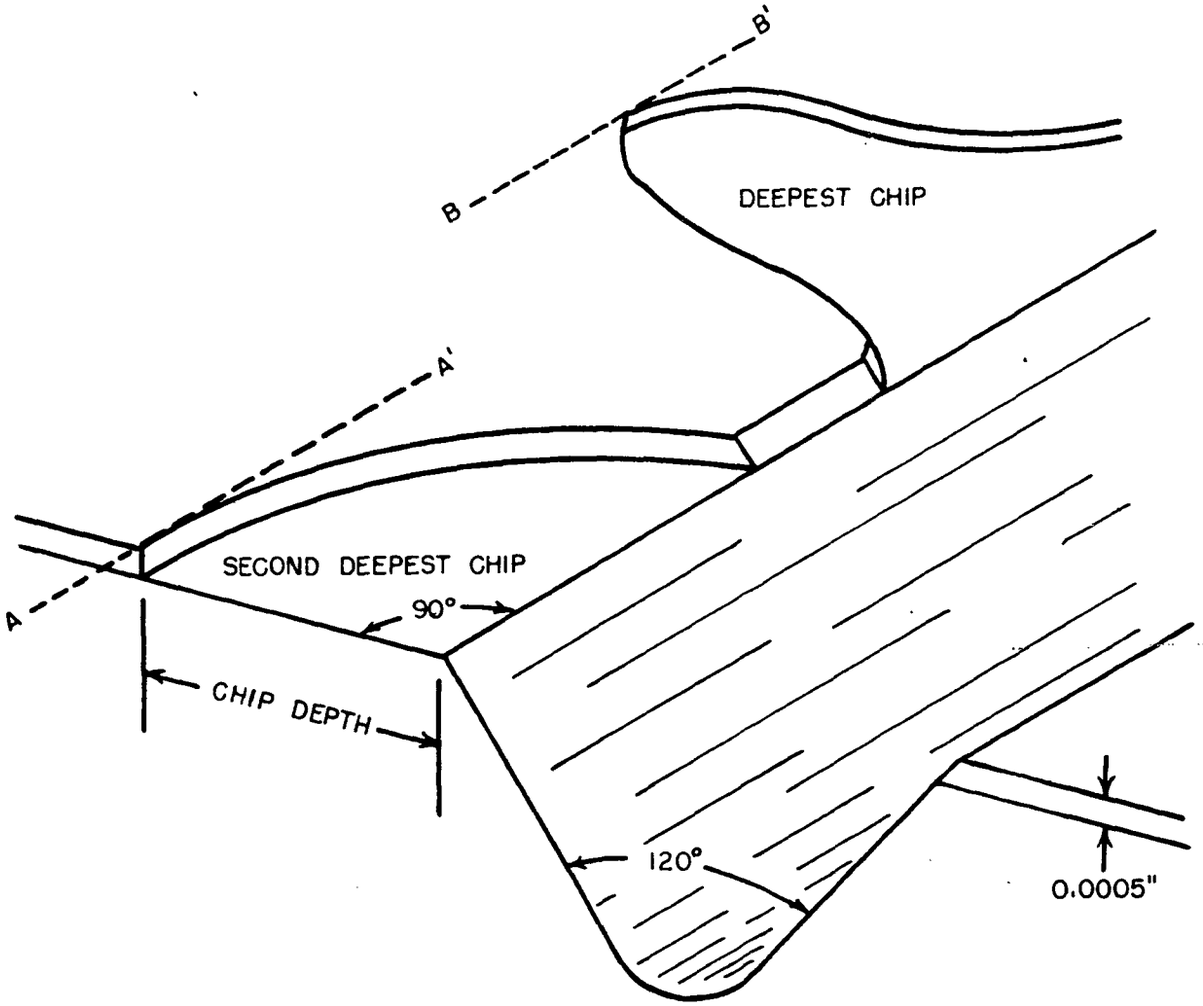


FIGURE 4

MEASUREMENT OF CHIP DEPTH (NOT TO SCALE)

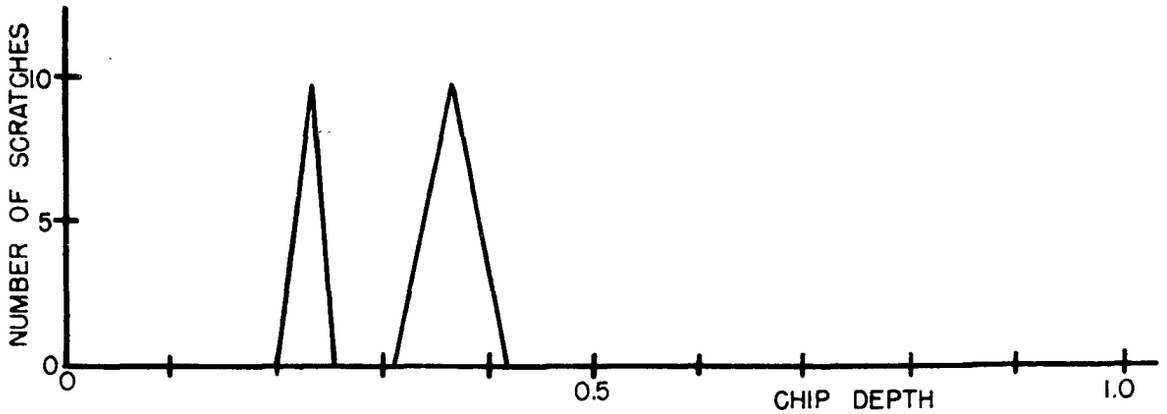


FIGURE 5 MEASUREMENTS MADE USING 15 CHIP DEPTHS PER SCRATCH

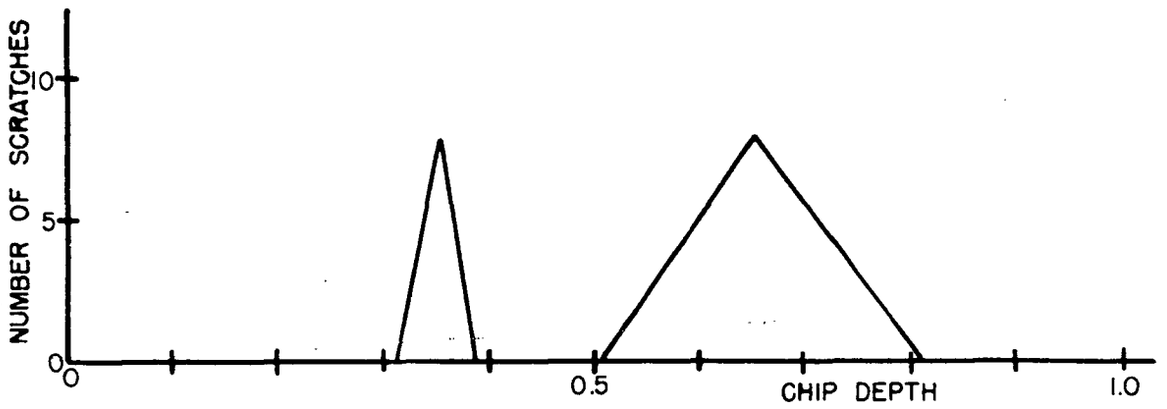


FIGURE 6 MEASUREMENTS MADE USING 3 CHIP DEPTHS PER SCRATCH

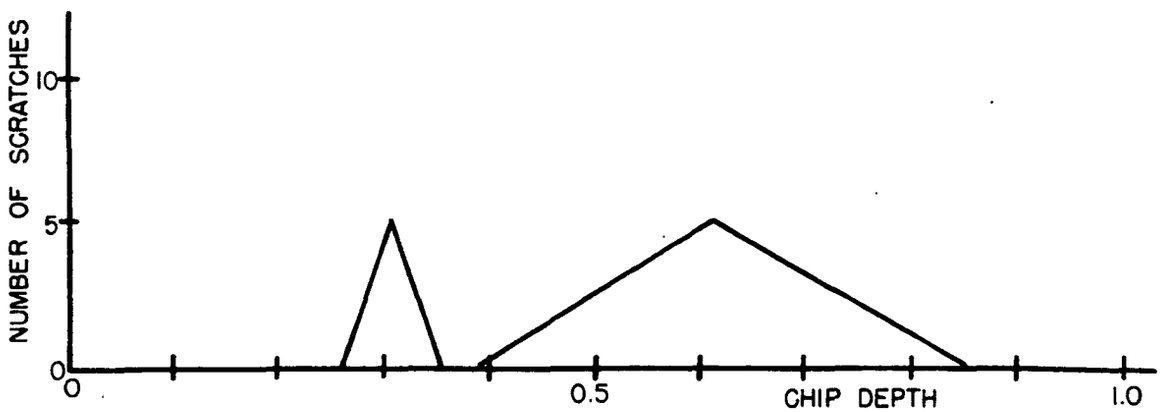


FIGURE 7 MEASUREMENTS MADE USING THE DEPTH OF THE SECOND LARGEST CHIP ALONG A SCRATCH

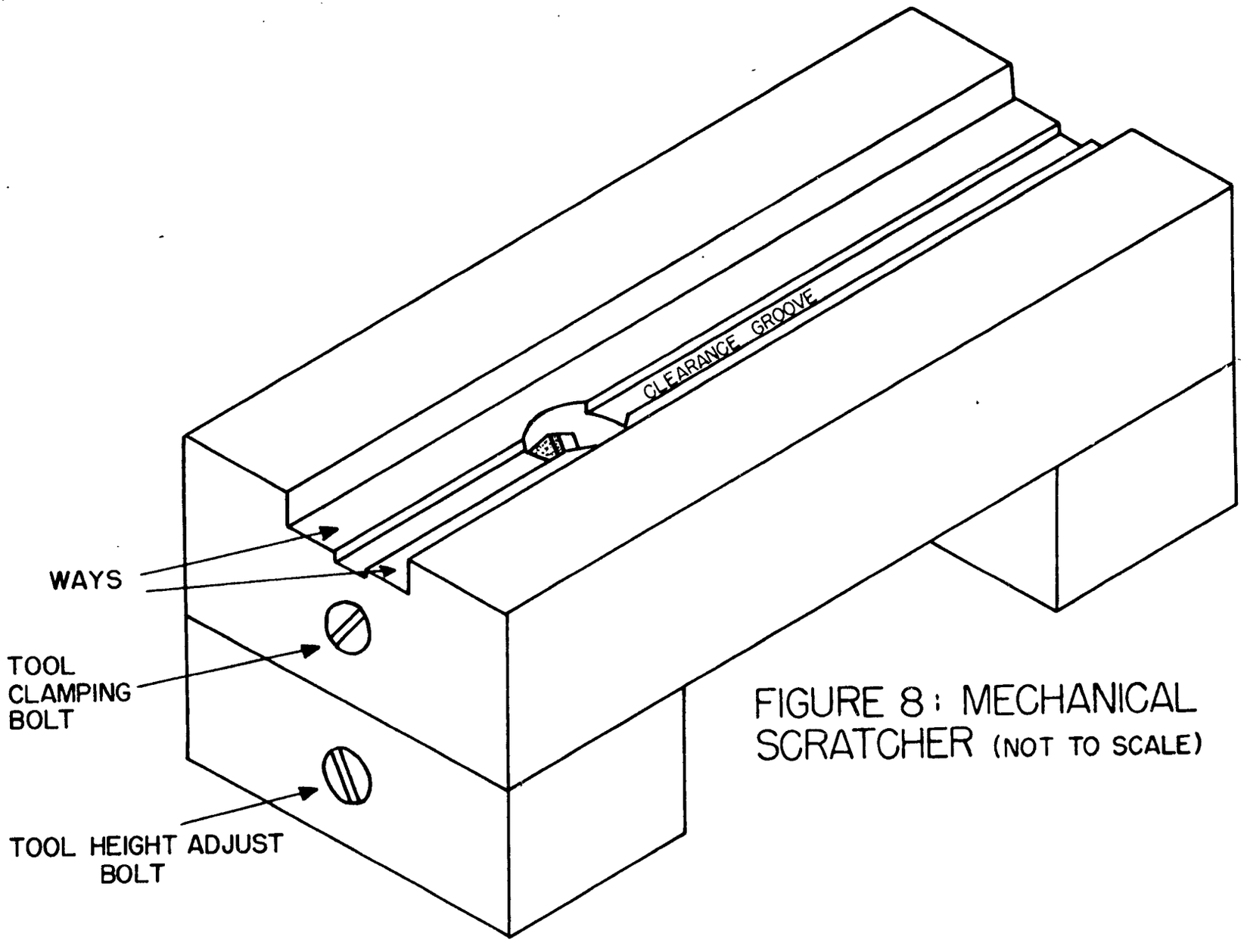


FIGURE 8: MECHANICAL SCRATCHER (NOT TO SCALE)

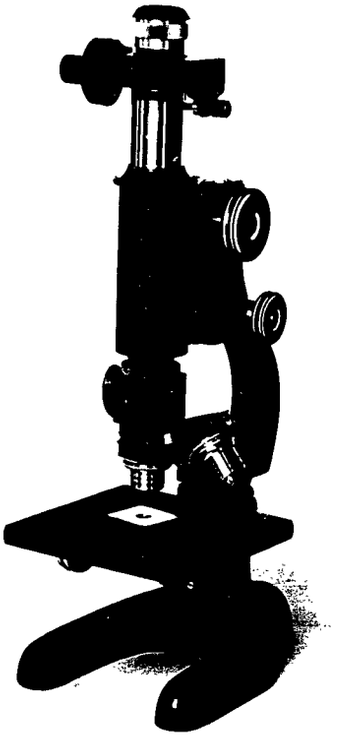


Figure 9: The Microscope.

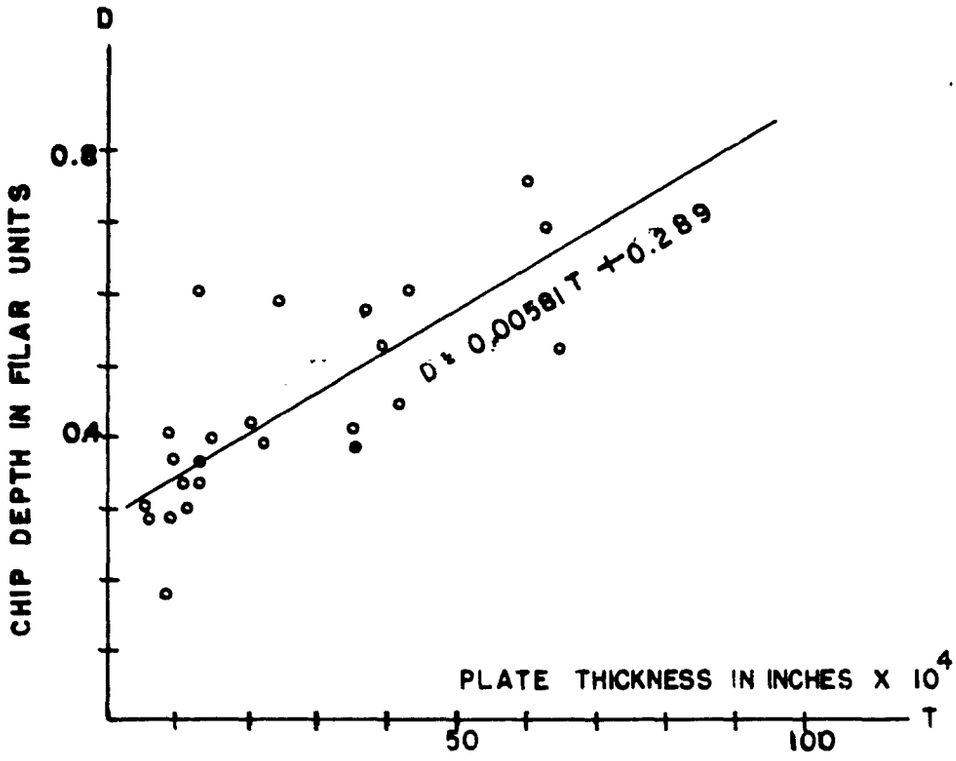


FIGURE 10: CHIP DEPTH VS PLATE THICKNESS.

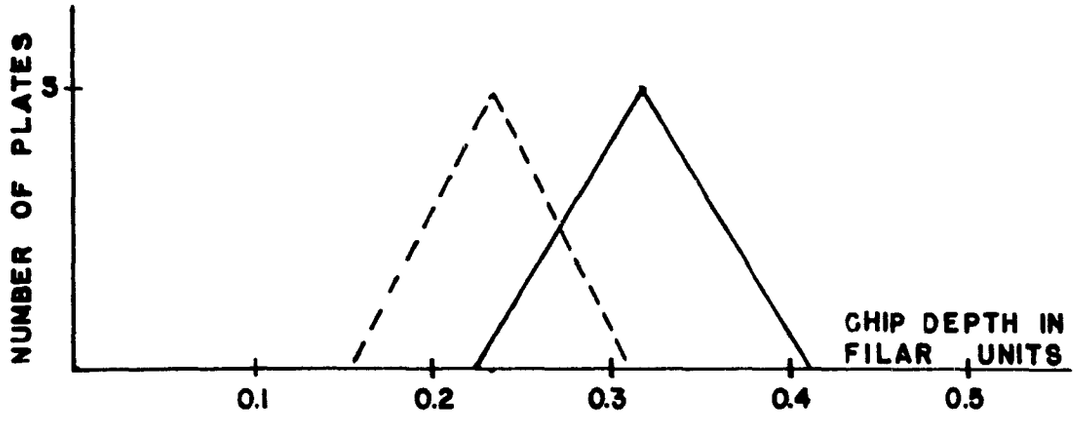


FIGURE 11: SCRATCH TEST RESULTS OF A GROUP OF SPECIMENS PLATED IN A UNICHROME BATH (SOLID LINE) AND A GROUP PLATED IN A STANDARD CHROMIUM BATH (DASHED LINE).

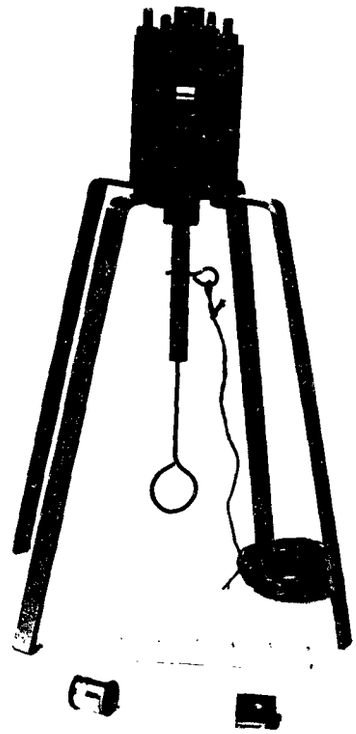


Figure 12: The Blast Test Apparatus.

Figure 13: Blast Test Specimen.

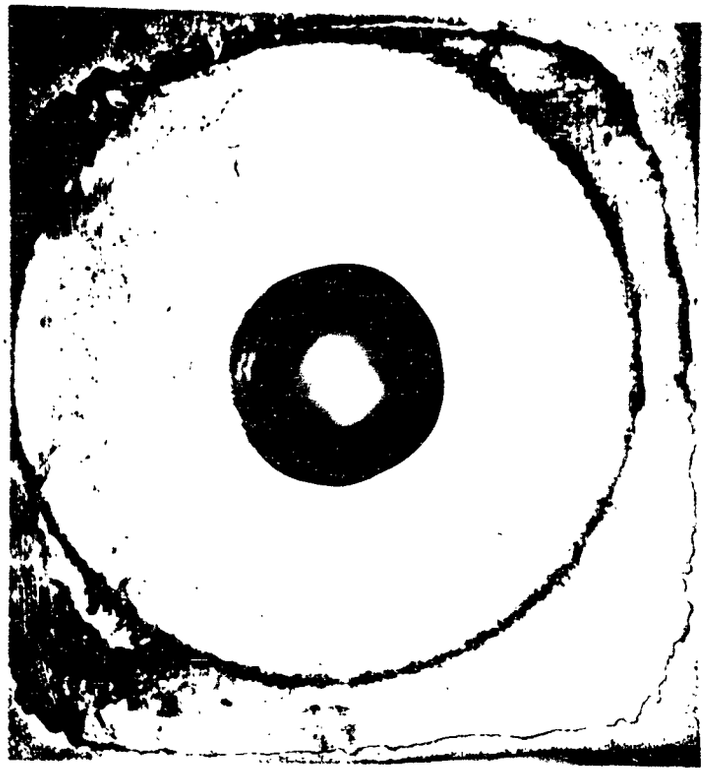


Figure 14: Blast Test Specimen.

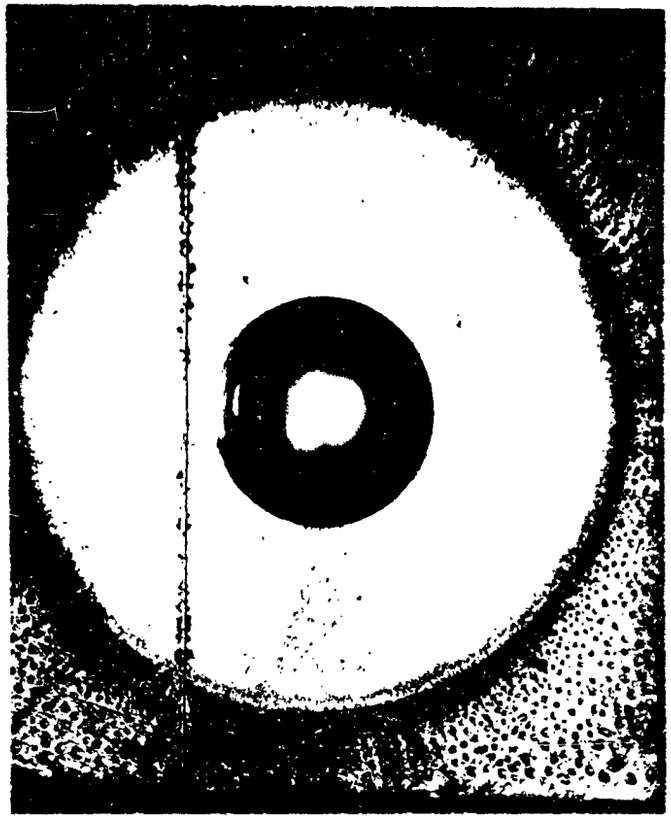


Figure 15: Blast Test Specimen.

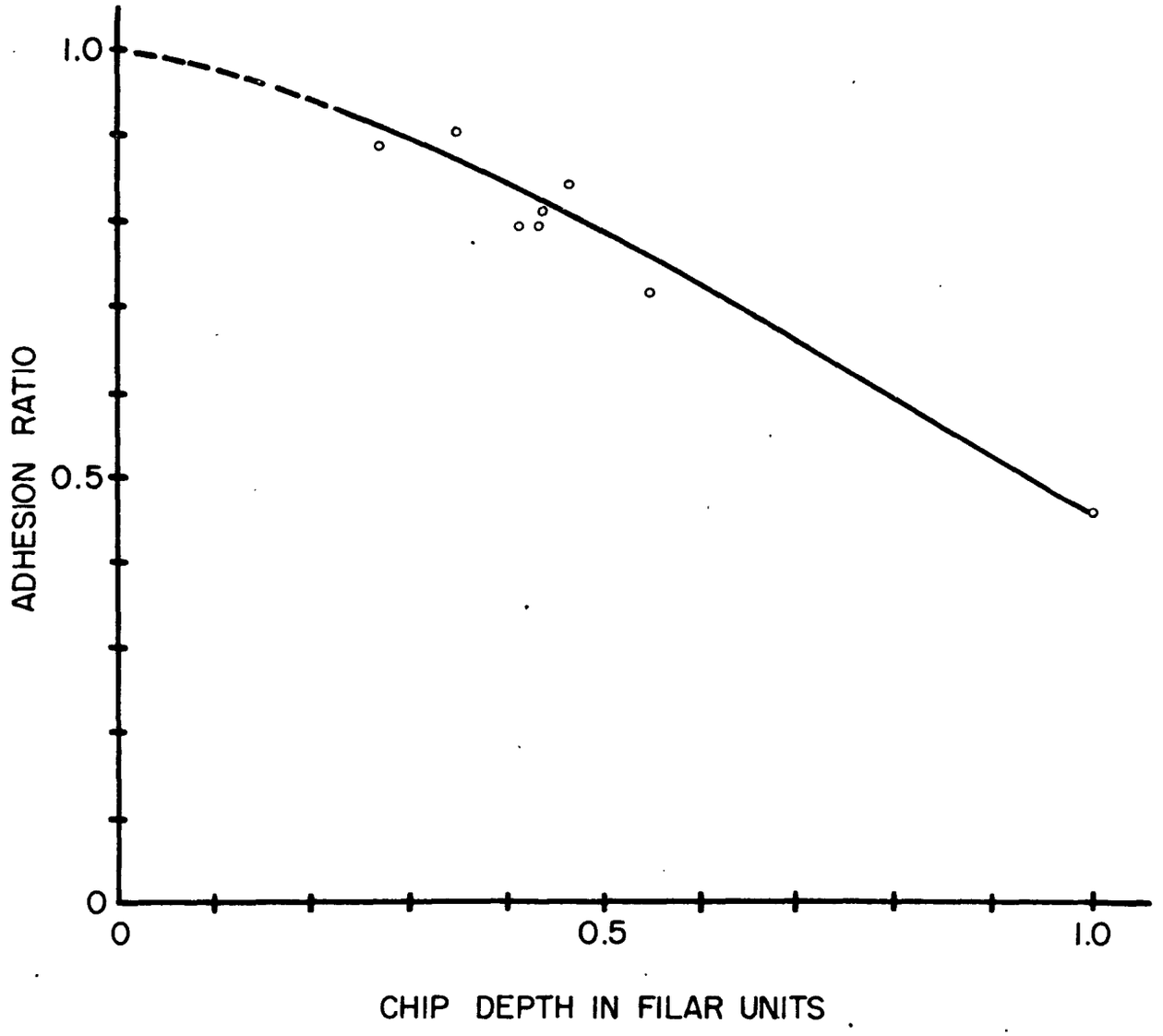


FIGURE 16: BLAST TEST ADHESION RATIO VS. CHIP DEPTH

THE SPECIMEN WAS CUT FROM A PIECE OF SHEET METAL, OF THE DIMENSIONS GIVEN IN THE UPPER FIGURE. IT WAS THEN BENT TO THE SEMICYLINDRICAL SHAPE SHOWN IN THE LOWER FIGURE. THESE FIGURES ARE NOT DRAWN TO SCALE.

THE SURFACE TO BE PLATED IS THE INNER CURVED SURFACE, BETWEEN THE LINES AA' AND BB', ONLY. THE RADIUS OF CURVATURE IS 1 INCH.

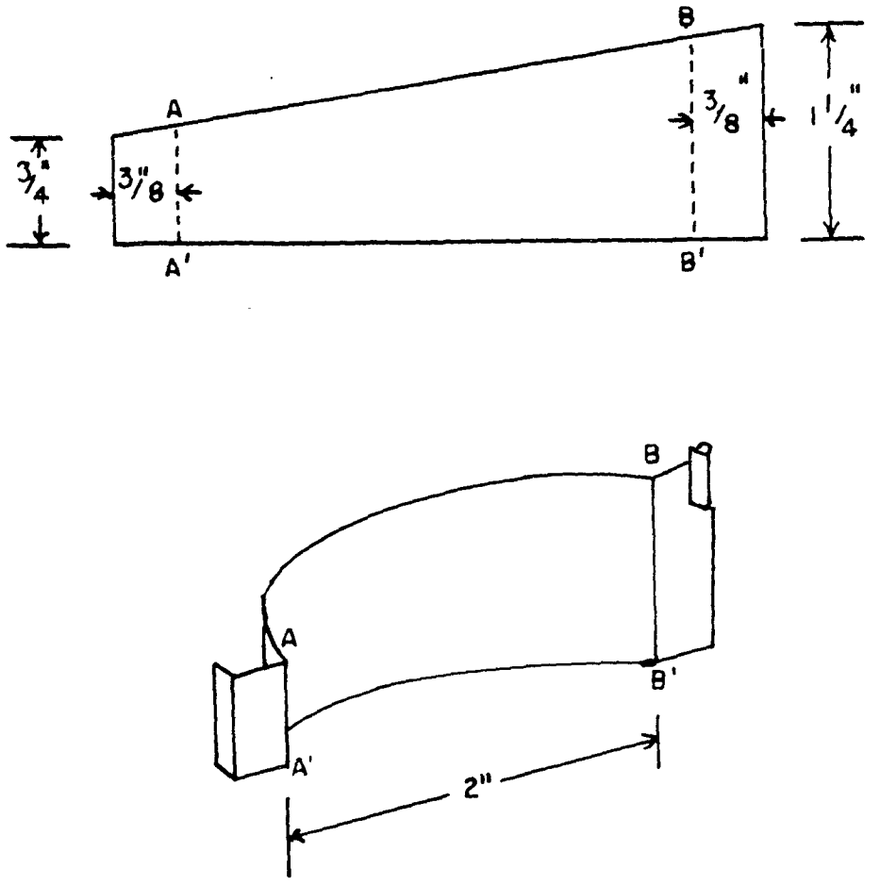


FIGURE 17: CATHODE DIMENSIONS FOR THE QUALIFICATION TEST.

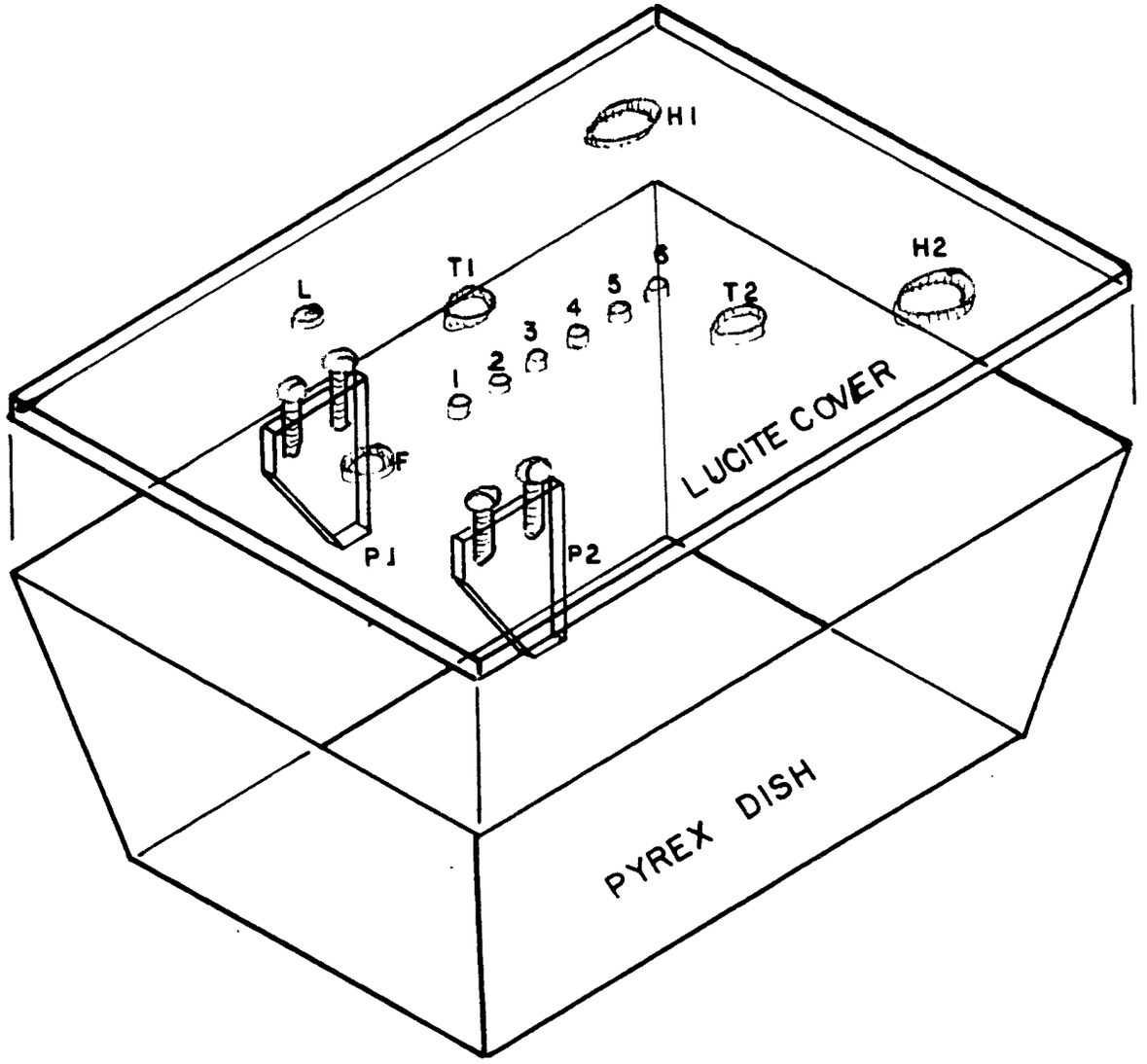
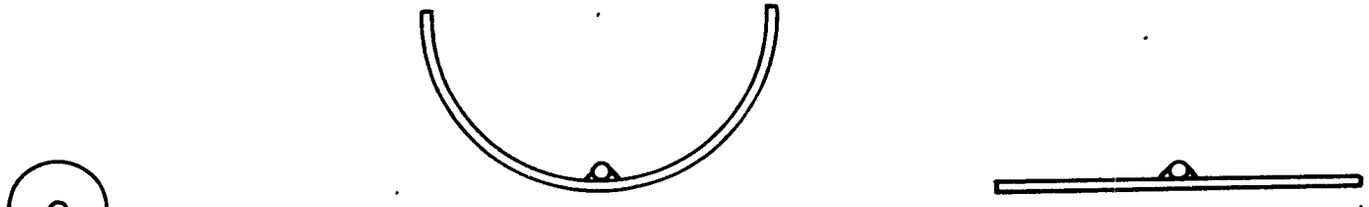
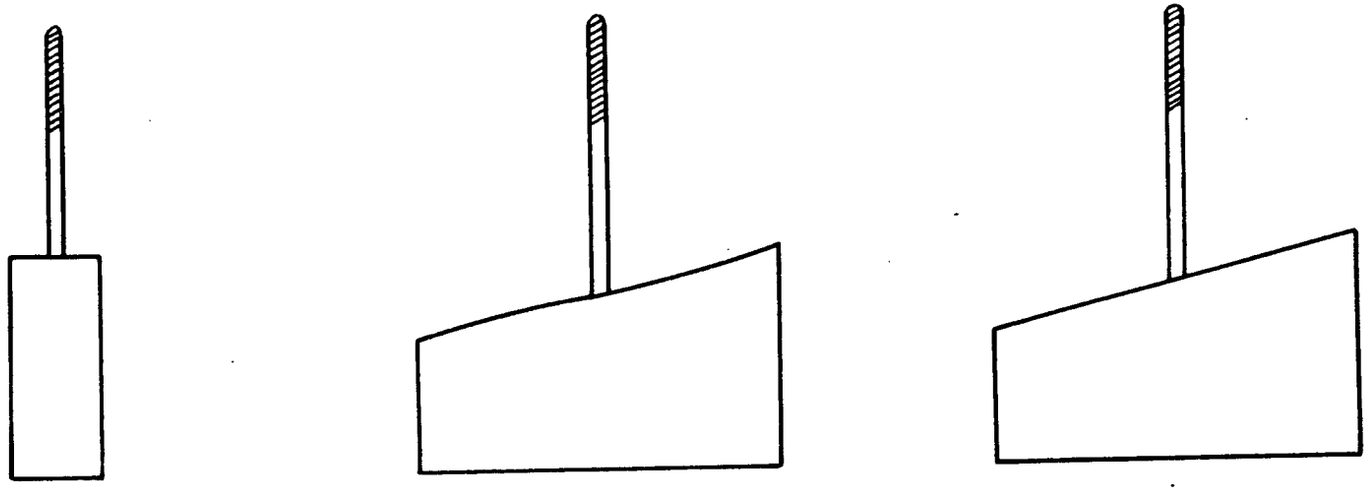


FIGURE 18: PLATING BATH AND EQUIPMENT POSITIONING COVER.



TOP VIEWS



ANODE 1 ANODE 2 ANODE 3

FRONT VIEWS

FIGURE 19: CHOICE OF ANODES FOR QUALIFICATION TEST FOR CHROMIUM PLATERS

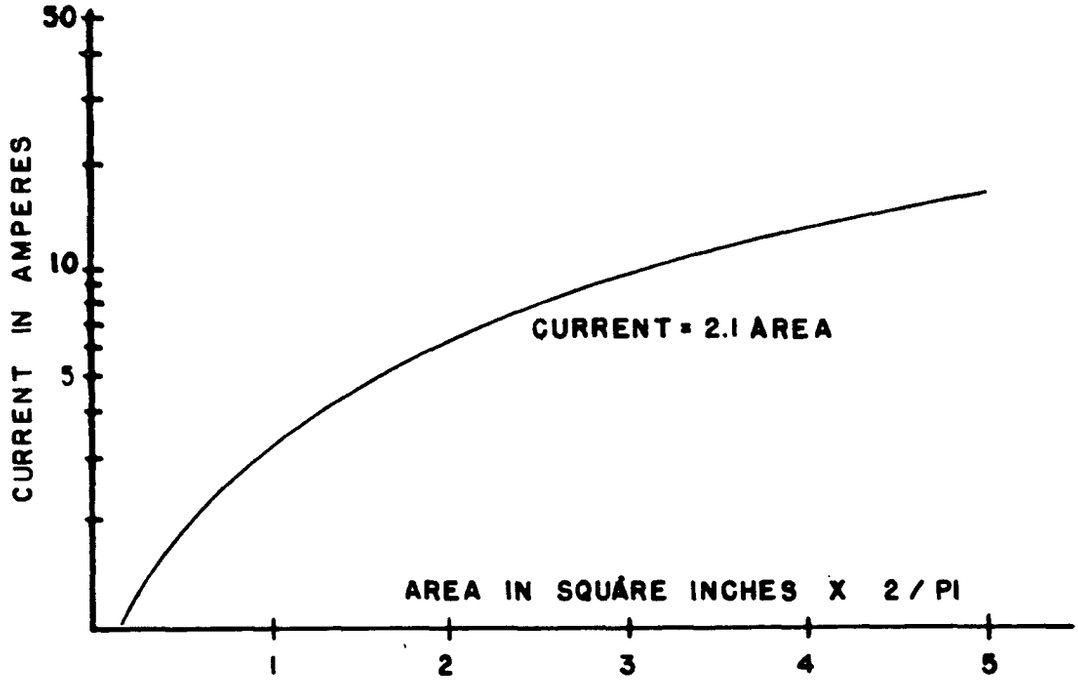


FIGURE 20: GRAPH OF PLATING CURRENT VS SPECIMEN SURFACE AREA

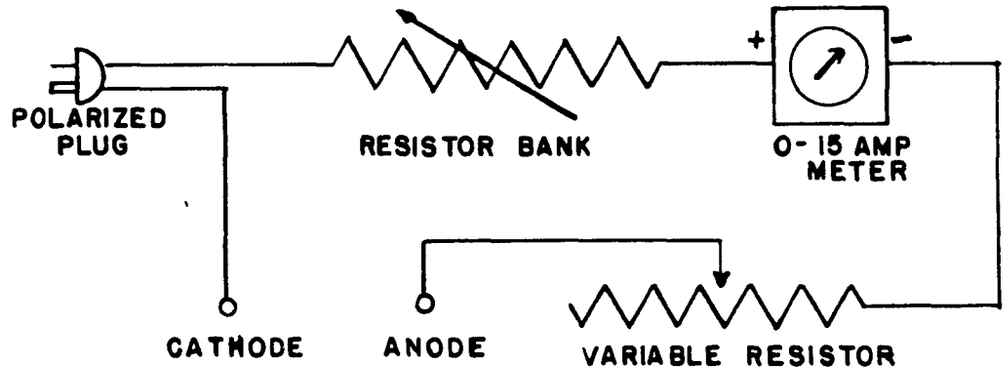


FIGURE 21: PLATING CUIRCUIT FOR QUALIFICATION TEST

APPENDIX A

SPECIFICATIONS FOR THE APPLICATION OF THE SCRATCH TEST

Scope:

This method covers the procedures for testing the adhesion of electroplated chromium on steel.

Choice of Test Specimens:

If possible, a section of the plated article itself should be chosen for the test. If this cannot be accomplished without serious damage to the article, a test specimen must be attached to the article, before cleaning and plating, so that the specimen and article will be treated simultaneously.

If an auxiliary test specimen is to be used, its metallurgical condition should be the same as that of the article and its chemical composition should be the same as that of the article.

The thickness of the plate on the section to be tested should be uniform and controlled within ±5% of the thickness of the standard plates, to which the test results will be compared.

For each article to be tested, at least five areas should be selected for testing, or the attached specimen should be large enough to permit testing at five different locations. The scratches should be separated by at least one inch from each other.

Specimen Preparation:

The surface of the specimen should be ground and polished to, at least, the same degree as the surface of the article being represented. The grinding operation should leave the same amount of distorted metal on the surface of the specimen as is on the surface of the article, prior to plating.

Scratching the Specimen:

The area to be tested should be scratched uniformly and evenly. The scratch should be from 3/8 to 3/4 inch long. At the level of the boundary of the plate to base metal, the side of the scratch should be straight.

The recommended scratching material is cemented tungsten carbide. This material is available in many shapes. any other material, equally hard and tough may be used. The point of the tool should be ground so that the included angle between the sides of the point should be 120° ± 5°. The tool may be ground with a 5 to 10° rake. The radius of curvature of the point should be sufficiently small so that when the scratch is made, the curved portion of the scratching tool will scratch well below the boundary of plate to base metal.

Plates thinner than 0.001 inches may be scratched by hand. Thicker plates should be scratched mechanically.

If, for any reason, a scratch was not made sufficiently deep, uniform, or straight, then a new scratch should be made at another position, at least 0.1 inches away from the first scratch.

Etching the Scratch:

The following reagent is recommended for etching the scratched area, although it is not absolutely necessary. This reagent will darken the steel, without affecting the chromium.

Nitric Acid (concentrated)	5 volumes
Ethyl Alcohol (95%)	95 volumes

Measurement of Chip Depth:

A metallurgical microscope should be used, with a filar micrometer eyepiece. The magnification should be from 100 to 150 diameters. The eyepiece need not be calibrated if the same microscope can be used for all measurements.

The depth of the second deepest chip along either edge of the scratch, should be measured. This chip should be selected by judgement of the relative depths of the chips that are found. The chip depth is the distance from the boundary

of plate to base metal, at the edge of the scratch, to the point of the chip that is the furthest from this boundary.

Standard Plates:

The standard plates are plates, of the same base metal as the specimens or articles to be tested, whose test results should serve as a basis, for comparison, of future test results.

These plates should be prepared under carefully controlled conditions so that there will be no doubt about their quality. Then they should be scratched and measured as outlined for the test specimens.

At least 50 standard plates should be made.

Interpretation of Results:

From the results of measurements of the chip depths of the standard plates, boundaries must be set for the determination of whether or not the adhesion of a plated article is acceptable. Therefore, if the result of a test should fall within the established boundary, the plate should be accepted, and if the result is not within the prescribed limits, the plate should be rejected and the article replated.

APPENDIX B

A SUGGESTED PROCEDURE FOR
ACCREDITING INDIVIDUAL PLATERS

Object:

A curved specimen is to be given a hard, adherent plate of chromium. The plating time is 30 minutes, which will produce a plate thickness of approximately 0.0005 inch, using the proper conditions.

The specimen that is provided is covered with a protective coating of zinc, which must be stripped. A copper lead wire must be attached to the specimen. All but the area to be plated should be masked with Duco, or airplane, cement. Then the specimen should be plated using the proper anode and proper operating conditions. After the 30 minutes plating time has been completed, or after the allowed test time has elapsed, the current should be stopped, and the specimen removed and cleaned.

On the questionaire, there are several questions that can be answered only after completion of the test: please answer them to the best of your ability. There are also several short questions to be answered during the test: these concern the operating conditions that you have chosen.

After completing the test and questionaire, you will be paid for your participation. Please sign the receipt on the questionaire, then, if you need more paper for your answers, use the back of the questionaire and scratch paper, but SIGN ALL THE SHEETS YOU USE.

You are allowed 90 minutes to complete the actual plating operation. Remember that a plating time of 30 minutes is required, and that 15 minutes may be needed to adjust the bath temperature prior to plating.

Along with this instruction sheet, you will be given a description of the apparatus and the questionaire. On the two resistor banks you will find a drawing which shows just exactly the area to be plated, a list of the specific conditions for chromium plating, and a graph to help you calculate the proper current. Please do not mark the figures.

Thank you very much for your cooperation and assistance.

LABORATORY FACILITIES

Plating Source:

The plating current is taken from the wall outlet, which is a supply of 115 volts, D. C. The resistor bank is used to regulate the current. The numbers marked on each switch indicate the approximate current that will pass when the switch is turned on. To obtain the desired current, turn on any combination of switches to give approximately the correct current, and then adjust the current critically by means of the slide on the water-cooled variable resistor that

is attached to the top of the resistor bank. A steady flow of water MUST be maintained through this variable resistor.

Except for clipping the leads on the electrodes, the wiring has been done.

The polarity of the leads may be found on the ammeter. None of the polarity markings on the clips or wall outlets should be followed because they may not be correct.

Bath Heater:

The plating bath is heated by a coil of nichrome resistance wire in a glass tube. This tube is formed so that the ends may project through two holes in the plating bath cover. There are two clips attached to a plug which may be plugged into the A.C. wall socket: these may be clipped onto the wires on the heater stems. The solution in the bath may be agitated by raising and lowering the heater tube. CAUTION: The heater stems may be hot.

Solutions:

The following chemicals are provided:

(1) The plating solution of the proper concentration.

(2) The dilute HCl for stripping the zinc.

(3) Oil.

(4) Cement for masking the surfaces that are not to be plated.

Plating Jig:

The plating jig consists of a Pyrex dish with a Lucite cover to hold the specimen. The specimen is already in the position that it should be in for the actual plating. It may be removed for the preparative operations. The small holes in the Lucite are for purposes of mounting the anode. The large hole, which is the farthest from the heater stem holes, is for the insertion of the tube from the aspirator, which will draw off the fumes.

Accessories:

These are items such as pliers, thermometer, towels, and labcoat that are available to aid in the work.